



Article

Alkaloids of *Solanum xanthocarpum* Stem as Green Inhibitor for Mild Steel Corrosion in One Molar Sulphuric Acid Solution

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Abstract: The residual ions of the acid cleaning processes induce the further corrosion of the metals, and this could be minimized using green inhibitors. Alkaloids extracted from plant parts could be cost effective and efficient inhibitors. In this work, alkaloids from *Solanum xanthocarpum* stem were successfully extracted, and they were characterized by qualitative chemical tests and spectroscopic measurements. As-extracted alkaloids were employed as green corrosion inhibitors for mild steel. The effectiveness of the inhibitor was determined by the weight loss and electrochemical measurement methods. From the weight loss measurement, the maximum inhibition efficiency of 93.14% was achieved. The temperature effect study revealed that the inhibitor can work up to a temperature of 58 °C. This could be one of the highest working temperatures among the reported green inhibitors. The electrochemical measurement reveals that the alkaloids could inhibit effectively up to 98.14% of the corrosion and serve as a mixed-type green inhibitor. A study on the kinetic parameters reflects that the inhibitor forms a potential barrier for the protection of a mild steel surface against corrosion. The values obtained from the thermodynamic parameters study reflect that the process is a spontaneous endothermic process. Based on the findings, it is revealed that the alkaloids extracted from *S. xanthocarpum* can serve as an excellent, eco-friendly and a promising green inhibitor against mild steel corrosion.

Keywords: *Solanum xanthocarpum*; alkaloids; green inhibitors; mild steel; weight loss; polarization



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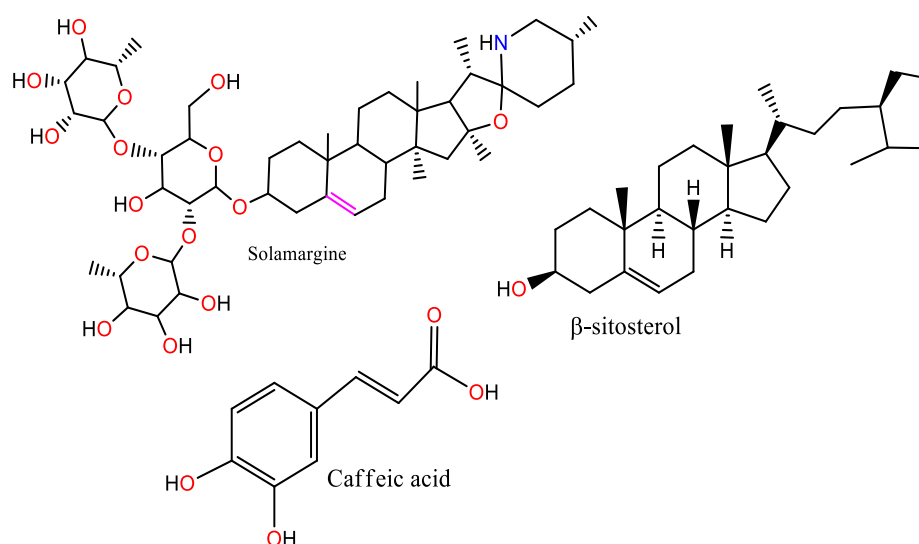
1. Introduction

Corrosion is an all-too-common result of the electrochemical reactions between metals with their environment, which results into the depletion of the resources, the untimely failure of the manufacturing plants, equipment and their components via physicochemical deterioration [1,2]. It is a spontaneous phenomenon known for having a destructive effect on the building materials composed of alloys, metals and steels [2–6]. Based on the carbon content (%) in the steel, it can be classified into pure iron ($\leq 0.04\%$), low carbon steel or mild steel (0.04–0.25%), medium carbon steel (0.25–0.60%), high carbon steel (0.60–1.25%), ultra high carbon steel (1.25–2.0%) and pig iron ($\geq 2.0\%$). Among these different forms of steel, mild steel is used extensively in the various construction sectors because of its low cost and pertinent properties [7]. Mild steel (MS) is a low carbon steel with a low tensile strength, typically with more ductility, machinability and weldability [8]. Because of these characteristics features, MS is the material of choice for various applications including structural steels, automobiles, decorations, fencing and nails in most of the chemical industries [9,10]. Despite their wide degree of applications, MSs are more prone to corrosion due to their thermodynamics instability, especially in acidic media and hence, the study of mild steel corrosion phenomena has become important, particularly in acidic media [11,12].

The damage caused by corrosion cannot be prevented completely, but the cost of the corrosion of the material can be reduced significantly using corrosion control methods [3,13]. The selection of materials, the use of coatings or painting [2,6,14], cathodic protection [15,16] and anodic protection are some common practices for the corrosion inhibition of mild steel. Nevertheless, these methods cannot be applied for the cleaning solutions.

Different acid solutions of high concentration employed in various industrial processes such as acid pickling, descaling, the cleaning of the boiler, the acidization of the oil well, and petrochemical processes not only removes the corrosion products from the surface, but it also causes the corrosion of mild steel [17]. Researchers of corrosion inhibition are concentrating on natural products as inhibitors rather than synthetic chemical inhibitors due to the effectiveness of the corrosion inhibitor at a low concentration, the facile method of application, their low cost and their eco-friendliness [18,19].

The corrosion inhibition performances of the plant extract are linked with the presence of organic compounds such as alkaloids, flavonoids, polyphenol, tannins, nitrogen bases, phenolics, carbohydrates, protein as well as hydrolysis products, etc. (Scheme 1) [20,21]. These metabolites usually bear a polar functional group containing nitrogen, sulfur or oxygen as heteroatoms, as well as triple or double conjugate bonds which act as major absorption centers on the surface of the metal. The lone pairs of electrons in the heteroatoms or π -electrons in the conjugate bonds are responsible for the formation of the layer over the steel materials [21,22].



Scheme 1. Structure of alkaloids, polyphenols and organic acids responsible for corrosion inhibition.

Several studies on green corrosion inhibitors for MSs have been reported. Most of the reported findings are based on the crude extract (methanol extract), and only a little research has been performed based on the alkaloid extracts. The ability of *Ochrosia oppositifolia* leaf extract was investigated to inhibit the corrosion of mild steel in an acidic medium. The isoreserpiline was the main alkaloid isolated from the extract [23]. Besides this, some recent works on green inhibition using plant extracts are listed in Table 1.

Table 1. Plants and their alkaloids as green corrosion inhibitors.

S.N.	Plant Name	Plant Parts	Medium	Method	Efficiency, %	Reference
1.	<i>Caulerpa racemosa</i>	Alga alkaloids	1 M HCl	Weight loss Polarization EIS	83 80 85	[24]
2.	<i>Ochrosia oppositifolia</i>	Bark and leaves alkaloids	1 M HCl	Polarization EIS	94 89	[25]
3.	<i>Neolamarckia cadamba</i>	Bark and leaves alkaloids	1 M HCl	Polarization EIS	89 83	[23]
4.	<i>Garcinia kola</i>	Seed alkaloids	5 M H ₂ SO ₄	Hydrogen evolution Polarization	99.4 98	[26]
5.	<i>Solanum melongena</i>	Leaves alkaloids	Alkaline medium Sodium trioxocarbonate	Weight loss	81.1	[27]
6.	<i>Geissospermum laeve</i>	Whole plant	1 M HCl	Polarization	92	[28]
7.	<i>Retama monosperma</i>	Seed alkaloids	1 M HCl	EIS	94.4	[29]
8.	<i>Fauvolfia macrophylla</i>	Bark	0.5 M H ₂ SO ₄ 1 M HCl	EIS Polarization	93, 97 92, 93	[30]
9.	<i>Retama monosperma</i>	Seeds alkaloids	1 M HCl	Electrochemical	94.42	[31]
10.	<i>Rhynchosstylus retusa</i>	Rhizome alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	87.51 93.24	[13]
11.	<i>Artemisia vulragis</i>	Stem alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	92.58 88.06	[12]
12.	<i>Solanum tuberosum</i>	Stem alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	90.79 83.22	[12]
13.	<i>Alnus nepalensis</i>	Bark alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	71.94 78.48	[32]
14.	<i>Acacia catechu</i>	Bark Alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	93.96 98.54	[7]
15.	<i>Coriaria nepalensis</i>	Stem Alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	96.41 97.03	[8]

In this work, corrosion inhibition on MSs in 1 M sulphuric acid media by using a green corrosion inhibitor extracted from *Solanum xanthocarpum* stem is presented. *Solanum xanthocarpum* is commonly known as yellow-berried nightshade and Bhatkaro in Nepali. It is mostly found in dry places as a weed on roadsides and wastelands [33]. The plants' fruits, roots and stems are used for medicinal purposes [34]. It was reported that the fruits of *S. xanthocarpum* contain several steroidal alkaloids such as solanacarpine, solamargine and other constituents such as caffeic acid, diosgenin, campesterol, daucosterol, etc. The major alkaloid present in this plant is Khasianine (Figure 1), which capacitates to bind with the metal surface due to the presence of nitrogen and oxygen atoms. A detailed study on this plant resulted in the isolation of solasonine and solasodine, β -sitosterol and campesterol [34]. The bioactive chemical constituents found in the stem of this plant are anthroquinone, flavonoids, saponins, tannins and alkaloids [35].

Herein, it is hypothesized that the alkaloid extract from *S. xanthocarpum* stem could serve as a potent green inhibitor against mild steel corrosion in one molar sulphuric acid solution. The efficacy of corrosion inhibition has been monitored in terms of the weight loss measurement and potentiodynamic polarization methods. The nature of the green inhibitor adsorption process on the MS steel surface has been studied in terms of the Langmuir, Freundlich and Tempkin adsorption isotherms.

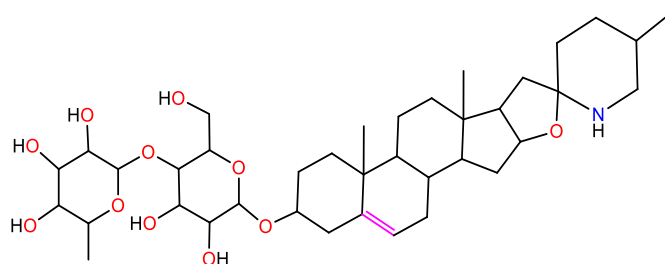


Figure 1. Structure of Khasianine alkaloid.

2. Materials and Methods

2.1. Chemicals and Instruments Used

The chemicals used in this experiment were all of the laboratory grades, and they were used as received without any additional purification. Sulfuric acid (Thermo Fischer Scientific, Waltham, MA, USA, 97%, sp. gr. 1.835), oxalic acid (Thermo Fischer Scientific, Waltham, MA, USA, 99%) and sodium hydroxide (Merck Life Science, Darmstadt, Germany, 97%) were used to prepare the corrosive media. Dichloromethane (Galaxo Laboratories, Greenford, UK, sp. gr. 1.326), methanol (Thermo Fischer Scientific, Waltham, MA, USA, sp. gr. 0.792), tartaric acid (RANBAXY Lab., Gurugram, India, 99.0%) and ammonia (Sisco Research Lab., Mumbai, India, sp. gr. 0.91) were used to extract the alkaloids. Instruments such as UV (Labotronics, Coventry, UK, LT-2808), FTIR (Perkin Elmer, Waltham, MA, USA, 10.6.2), a Rotary evaporator (IKA 10) and a Digital Vernier Caliper were used as analytical tools.

2.2. Extraction of Alkaloids from *S. xanthocarpum* Stem

The stem of the plant was collected from Dang (Latitude: 28.1545° N, Longitude: 82.3235° E), Lumbini Province, Nepal. The collected sample was shade dried, and the dried sample was ground into fine powder. One hundred g powder sample was soaked in 800 mL hexane for 24 h, which was followed by filtration. Then, 800 mL methanol was added in the residue, and it was stirred and allowed to stand for 15 days, and then, it was filtered. The filtrate was taken in a beaker and acidified with 5% tartaric acid until the pH reached 3.0. The alkaloids were precipitated in the form of salt. The mixture was then filtered. The residue was taken, and ammonium hydroxide solution was added to maintain a pH of 10. The alkaline solution was subjected to solvent extraction with an equal volume of dichloromethane (DCM). The DCM layer containing the alkaloids was collected in a beaker and concentrated using a rotary evaporator. The concentrated solution was then dried using a water bath at 40 °C until it was dry.

2.3. Chemical Test for Alkaloids

The *S. xanthocarpum* stem extract (SXSE) was subjected to the Mayer's test, Dragendorff's test and Wagner's test to ensure the presence of alkaloids. The spectra of the alkaloids extracted from the stem of the *Solanum xanthocarpum* plant were documented using a Perkin Elmer Spectrometer, the 10.6.2 version. The background correction was accomplished by using isopropanol. All of the spectral data were collected from a 4000–450 cm^{−1} cutoff range with a 4 cm^{−1} resolution.

2.4. Preparation of MS Specimen

Mild steel coupons of A516 with a composition of 0.21–0.27% C, 0.13–0.45% Mn, 0.79–1.30% P, 0.035% S and the rest of iron were used in this study. The coupons were prepared from an MS sheet sourced from the local supplier, Kathmandu, Nepal. Before each experiment, the coupons were mechanically abraded with SiC papers (P150) of different grits (100–1200) and stored in the moisture free desiccators. Prior to the experiment, each sample was sonicated in ethanol and dried.

2.5. Preparation of Inhibitor Solution

The inhibitor stock solution was prepared by dissolving 1 g of alkaloids in 1 L of one molar sulphuric acid solution, and it was labeled as 1000 ppm. The other required concentrations of the inhibitor solutions (200, 400, 600 and 800 ppm) were prepared by performing the serial dilution of this stock solution using one molar sulphuric acid. These solutions were stored at a low temperature.

2.6. Weight Loss Measurement Method

The weight loss measurements were performed by immersing the MS coupons into the acid solutions in the absence and presence of different concentrations of the inhibitor. Prior to each weight loss measurement, the dimension of each coupon was measured using a Vernier caliper. The initial weight of the sample was assigned as recorded from an electronic balance, and it was subsequently immersed in the acid solution. After 0.5 h immersion, the sample was taken out, washed thoroughly with distilled water, dried and reweighed. From the initial and final weights of the sample, the loss in weight was calculated.

Similar experiments were carried out for 1, 3, 6 and 24 h immersion times, separately, as well as for different concentrations of the inhibitors (200, 400, 600 and 800 ppm) for the mentioned times. The temperature's effect on the weight loss was studied, for which the immersion tests were carried out for 1 h at different temperatures (28, 38, 48 and 58 °C), separately. From these data, the inhibition efficiency and surface coverage by the inhibitors were calculated using the formula mentioned in Equations (1) and (2).

$$\text{Inhibition efficiency (\%)} = \frac{(W_2 - W_1)}{W_2} \times 100 \quad (1)$$

$$\text{Surface coverage} = \frac{(W_2 - W_1)}{W_2} \quad (2)$$

where, W_1 and W_2 are the weight losses for the mild steel in the presence and absence of the inhibitor, respectively.

2.7. Electrochemical Measurement

The effectiveness of the inhibitor for mild steel corrosion was monitored by the electrochemical measurements. The open-circuit potential (OCP) and the potentiodynamic polarization were carried out using the Hokuto Denko potentiostat (HA-151, Tohoku, Japan) in a three-electrode system. The three-electrode cell set-up was maintained by using an MS coupon as a working electrode, graphite as a counter electrode, and saturated calomel electrode (SCE) as a reference electrode. Before the polarization, the equilibrium potential was achieved by measuring the OCP for 30 min. The potentiodynamic polarization of the specimen was measured just after the OCP recording in a three-electrode system. Both the anodic and cathodic polarization in the potential window from -0.8 to -0.2 V, i.e., ± 300 mV from the OCP with a scan rate of 60 mV/s and a maximum current of 10 mA, was applied for both the immersed and as-immersed conditions [12]. Inhibition efficiency was calculated using the formula in Equation (3):

$$\text{Corrosion inhibition efficiency (IE, \%)} = \frac{i_{\text{corr}} - i_{\text{corr}}^*}{i_{\text{corr}}} \times 100 \quad (3)$$

where, i_{corr} and i_{corr}^* are the current densities in absence and presence of inhibitor, respectively.

2.8. Adsorption Isotherm and Kinetic Study

In this work, the adsorption isotherms were studied by immersing the MS coupons into the inhibitor solutions of different concentrations at the laboratory temperature. To identify the adsorption process, the molecular interaction/thickness of the adsorbed layer onto the MS surface and the free energy of the adsorption, three different adsorption isotherms models (Langmuir, Freundlich and Tempkin adsorption isotherms) were studied.

The Langmuir adsorption isotherm was studied by immersing the MS coupons into the inhibitor solutions of different concentrations for 1, 6 and 24 h intervals to study whether the adsorption is monolayered or multilayered.

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (4)$$

The free energy of the adsorption was calculated according to the relation:

$$(\Delta G) = -RT \ln(55.5 K_{ads}) \quad (5)$$

The validity of Freundlich adsorption isotherm was assessed according to the following relation [12,13]:

$$\ln \theta = \frac{1}{n} \ln C + \ln K \quad (6)$$

Temkin adsorption isotherm was evaluated based on the following equation [15]:

$$\theta = -\frac{1}{2a} \ln C - \frac{1}{2a} \ln K \quad (7)$$

Activation energy of corrosion process was evaluated based on the following equation [36,37]:

$$\log(CR) = \log A - \frac{E_a}{2.303RT} \quad (8)$$

where A is the Arrhenius pre-exponential constant, and T is the absolute temperature. In all of these models, the linear correlation coefficient (R^2) values of the straight lines were used to assess the validity of the adsorption model.

The enthalpy and entropy of the system can be calculated according to the following equation [36–38]:

$$\log\left(\frac{CR}{T}\right) = \log \frac{R}{hN} + \frac{\Delta S^\circ}{2.303 R} - \frac{\Delta H^\circ}{2.303RT} \quad (9)$$

where h is the Planck's constant, 6.6261×10^{-34} Js, and N is Avogadro's number, $6.0225 \times 10^{23} \text{ mol}^{-1}$.

2.9. Surface Morphological Study

An optical microscopic image of the polished MSs as well the MS coupons immersed in the acid and 1000 ppm green inhibitor solutions were captured at Central Department of Chemistry, Tribhuvan University, Nepal. All of the images were captured using a high-definition optical microscope (RXLR-4, Radical Scientific, Ambala Cantt, India).

3. Results

The *Solanum xanthocarpum* stem alkaloids were extracted, and the DCM fraction was collected in a beaker and concentrated using a rotary evaporator. The concentrated solution was then dried at 40 °C until it was dry using a water bath. The extract was characterized by a chemical test as well as an FTIR spectroscopic test. The corrosion inhibitory behavior of the alkaloids was studied by the weight loss and electrochemical methods. In addition, the adsorption isotherm and kinetic study of the extract was carried out on the MS surface.

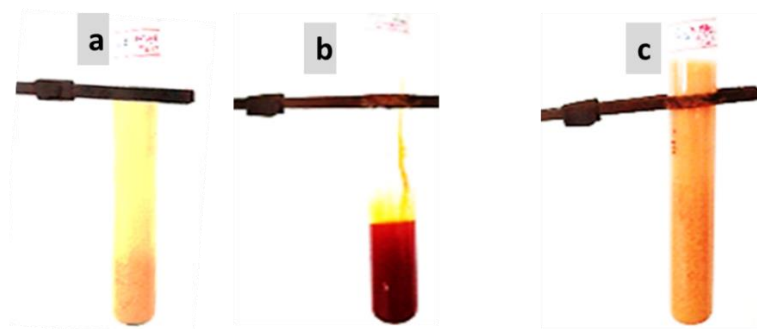
3.1. Characterization of Alkaloids

3.1.1. Chemical Test of Alkaloids

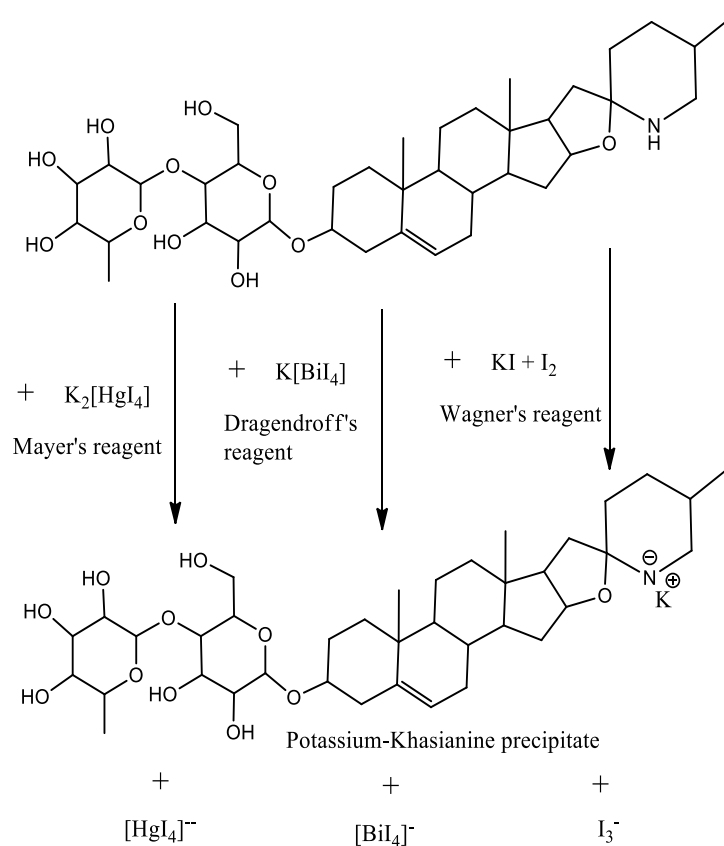
The successful extraction of the alkaloids from the stem of *Solanum xanthocarpum* was achieved by the chemical test methods. The results obtained are in Table 2. The precipitates that were formed with a characteristic color for each test are given in Figure 2.

Table 2. Chemical test for alkaloids.

S.N.	Experiment	Observation	Result	Inference
1.	Mayer's Test	Orange precipitate	Alkaloids presence	Positive
2.	Dragendroff's Test	Orange-red precipitate	Alkaloids presence	Positive
3.	Wagner's Test	Reddish brown precipitate	Alkaloids presence	Positive

**Figure 2.** Test for alkaloids obtained from *S. xanthocarpum*: (a) Mayer's, (b) Dragendroff's and (c) Wagner's tests.

The major alkaloid present in this plant is Khasianine. The reactions involved in the test for alkaloids are based on this Khasianine molecule, as given below (Figure 3) [13]

**Figure 3.** Reactions involved in the test for alkaloids based on this Khasianine molecule.

3.1.2. FTIR Spectroscopic Measurement

The type of bonding, π -bond conjugate system, functional group and aromatic and aliphatic structures present in the organic compounds were identified with the help of the FTIR spectra.

The recorded spectral lines are shown in Figure 4, and the recognition of the absorption bands is as follows. A broad absorption band observed in the range of 3500–3150 cm^{-1} indicates the presence of the N-H and O-H stretching groups. The presence of both the hydroxyl and amine groups in the molecule could impart a broad peak in the spectra. The peak observed at 2957 cm^{-1} means that there is C-H aliphatic asymmetry stretching vibration along with methyl and methylene groups. An absorption band observed in the range of 1540–1870 cm^{-1} with a peak at 1658 cm^{-1} indicates the presence of the carbonyl group. The peak observed at 1065 cm^{-1} is due to the C-N stretching of the amines. The peak observed at 576 cm^{-1} is due to the N-H bending vibration of the primary and secondary amines [12,32,39]. These findings support the presence of alkaloids and hydroxyl functional moieties in the stem extract of *S. xanthocarpum*.

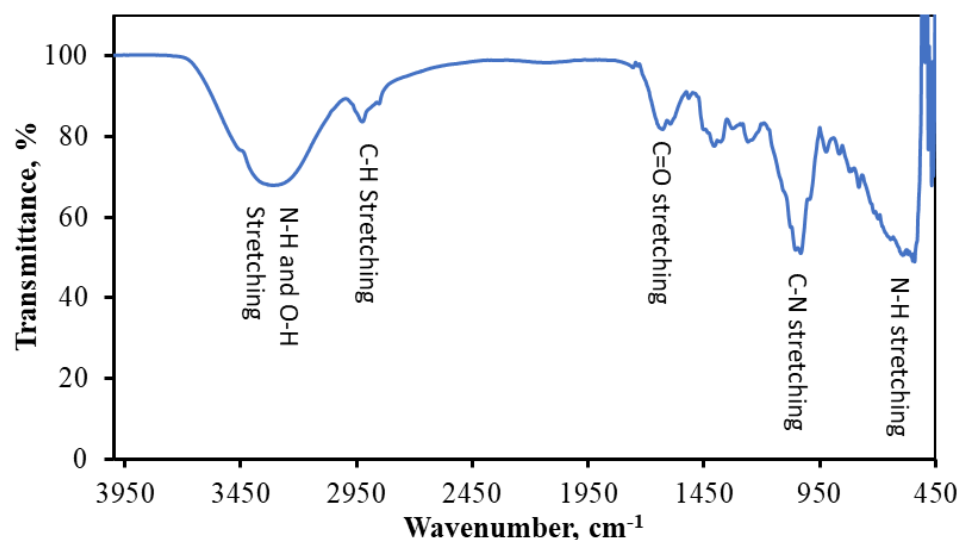


Figure 4. IR spectrum of alkaloid extract of *Solanum xanthocarpum*.

3.2. Findings from Immersion Tests

The immersion tests were performed to determine the inhibition efficiency of the alkaloids extracted from the *Solanum xanthocarpum* stem. The interaction of the MS with the surrounding aggressive environment upon immersion leads to the formation of corrosion products which give rise to the decrease in the weight of the MS. This decrease in weight is influenced by the nature of the surface, the working temperature and the concentration of the inhibitor solution.

3.2.1. Effect of Alkaloid Concentration

The interaction of a caustic medium with the MS surface results in the formation of corrosion products, consequently decreasing the weight of the MS. Addition of the inhibitor solutions can withstand the weight loss, and its efficacy of corrosion resistance depends on its concentration. In this experiment, inhibitor solutions of different concentrations (200, 400, 600, 800 and 1000 ppm) were examined against the MS corrosion. The weight loss is a lot higher for the MS that was immersed in the 200 ppm solution than it is for the rest of them, and it was found to be minimal for the sample that was immersed in the 800 and 1000 ppm solutions. This leads us to conclude that the 200 ppm inhibitor concentration is unable to control the MS corrosion for 24 h.

The loss in weight was very high for the MS that was immersed in the acid-only solution. The loss in weight of the MS gradually became smaller with an increase in the concentration of the inhibitors, as shown in Figure 5a. This may be due to the adsorption of inhibitor molecules onto the metal surface. The minimum weight loss value was observed in the 800 and 1000 ppm solutions after 0.5 h of immersion. The weight loss values for both the concentrations 800 and 1000 ppm was found to be similar for the 24 h immersed

sample, indicating that the optimum concentration of the inhibitor for corrosion inhibition is 800 ppm.

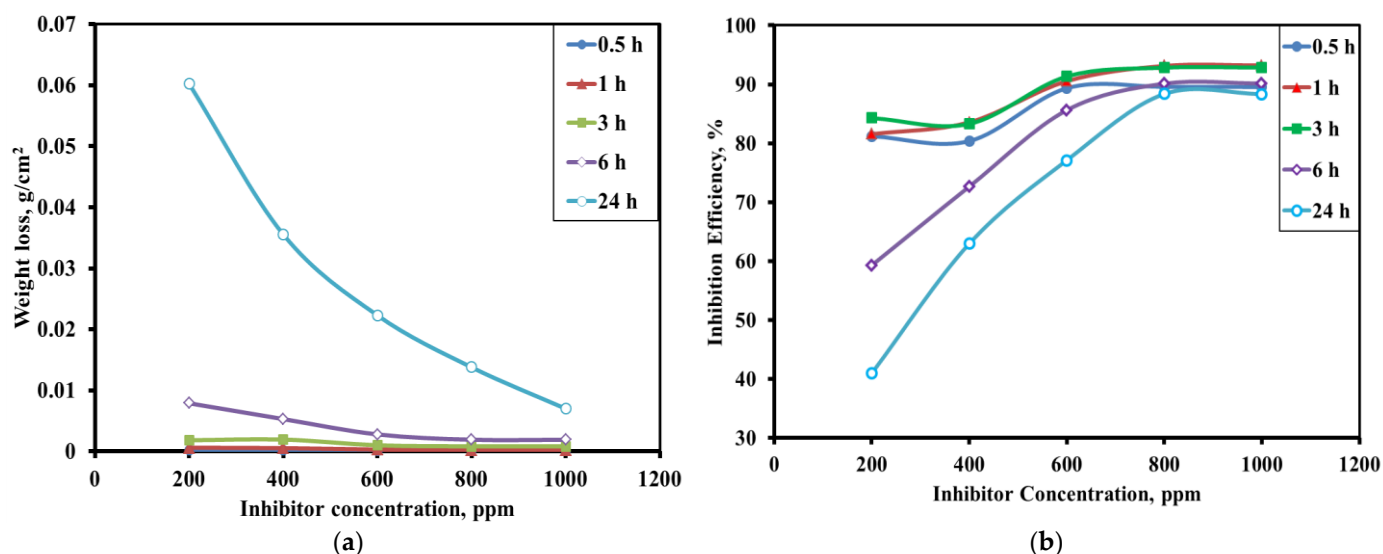


Figure 5. Effect of different concentrations of alkaloids on (a) weight loss and (b) inhibition efficiency for mild steel corrosion in 1 M H₂SO₄ solution at various immersion times, which were obtained by taking each triplicate measurement ($n = 3$).

The corresponding inhibition efficiency of the alkaloid with its concentration effect is shown in Figure 5b. In the study, the inhibition efficiency was found higher at a higher concentration of the inhibitor. The experiment was carried out with different concentrations (0, 200, 400, 600, 800 and 1000 ppm) of the inhibitor solutions in 1M H₂SO₄. The weight loss data indicate that the inhibition efficiency increases with the increase in the inhibitor concentration of up to a 600 ppm inhibitor concentration for the 0.5, 1, 3 and 6 h immersed samples, whereas it increases to up to 800 ppm for the 24 h immersed sample. This is because at higher concentrations, a large number of inhibitor molecules are available for adsorption onto the MS surface. The maximum inhibition efficiency is found to be 91.92% at a 3 h immersion time in the 800 ppm inhibitor concentration. As we further increased the inhibitor concentration, the efficiency remained constant, indicating that it had reached the saturation point. The optimum concentration of the inhibitor for lower immersion time is 600 ppm, but for the 24 h immersion time, it is 800 ppm. This efficiency did not remain constant as we increased the immersion time. The minimum efficiency of the 800 ppm inhibitor concentration was found to be 80.39% at 24 h. Afterwards, all of the weight loss experiments were performed up to an 800 ppm concentration as this is the optimum concentration of the inhibitor.

3.2.2. Effect of Immersion Time

The effect of immersion time on the weight loss of the MS was monitored at a laboratory temperature (28 °C). The effect of the immersion time on the corrosion inhibition was studied by performing weight loss experiments at different time intervals (0.5, 1, 3, 6 and 24 h). The weight loss data of the MS coupons immersed in acid in the absence and presence of inhibitor solutions for different immersion times are shown in Figure 6a.

The results obtained from this experiment showed that the presence of inhibitors in a caustic environment reduces the weight loss of the MS. The weight loss of the MS immersed in the acid-only solution is higher than that which occurred when it was immersed in presence of the inhibitor solutions. Initially, the weight loss was very small, but after a period of prolonged immersion, the weight loss of the MS became increased, and it had the same trend. The weight loss trend in this experiment suggests that the inhibitors could prevent and protect the MS surface from a caustic environment.

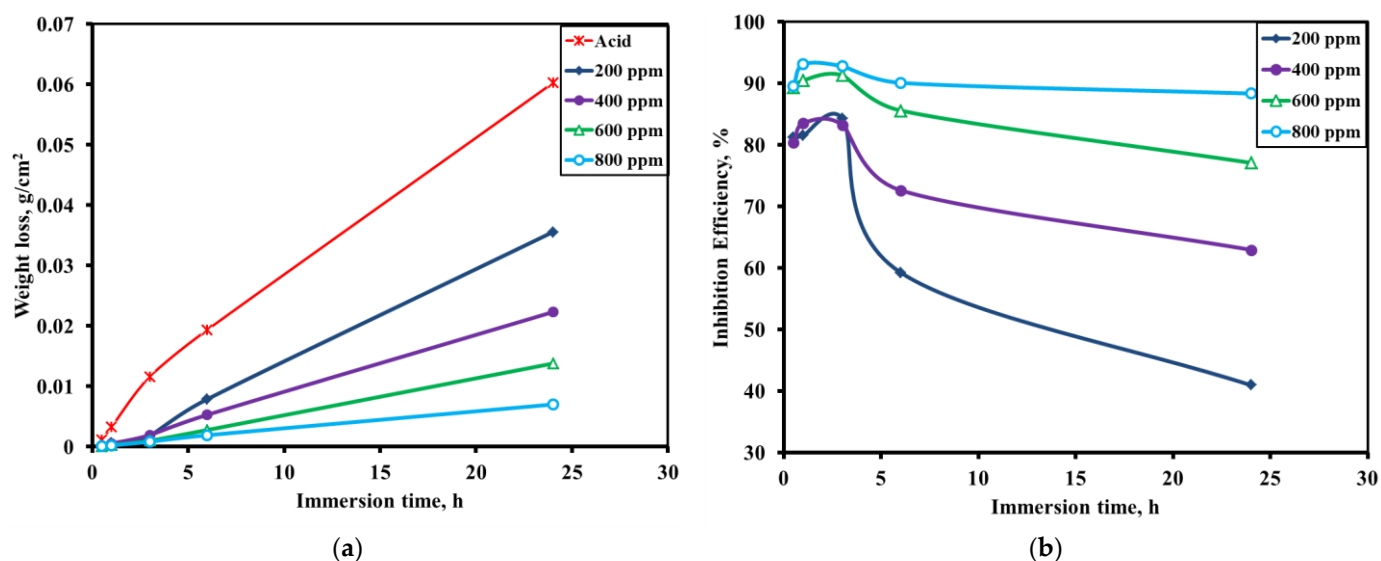


Figure 6. Effect of immersion time on the variation of (a) weight loss and (b) inhibition efficiency for the corrosion of MS dipped in acid and different concentrations of inhibitor solution at room temperature, which were obtained by taking triplicate measurements ($n = 3$).

The inhibition efficiency of the inhibitor at different immersion times (h) is shown in Figure 6b. It is demonstrated that the inhibition efficiency gradually increased up to the 3 h immersion time, but afterwards the inhibition efficiency remained almost constant for the 800 ppm inhibitor solution. However, the case is different for the 200 ppm inhibitor solution. In this case, the inhibition efficiency gradually decreased after the 3 h immersion time. This implies that during continuous immersion, the desorption of the inhibitor molecules from the MS surface as well as the formation of the Fe-inhibitor chelate complex could happen [12,32].

3.2.3. Effect of Temperature

Temperature greatly influences the corrosion rate as well as the inhibitor action. The temperature effect on the inhibition process was studied by varying the working temperature. The weight loss of the 1 h immersed MS sample in the inhibitor solutions of different concentrations (200, 400, 600 and 800 ppm) was measured at different working temperatures (28, 38, 48 and 58 °C). The corresponding inhibition efficiency was measured using references to the weight loss of the 1 h immersed MS sample in an acid-only solution. All of the observed data are shown in Figure 7a. It is demonstrated that the weight loss in the acid-only solution is very high in comparison to those of the others. The weight loss of the MS was also found to be temperature dependent. In the graph, it is clear that the weight loss of the MS became increased gradually with an increasing temperature, it is because the hydrogen evolution reaction takes place so rapidly at an increasing activation energy with a higher temperature.

The inhibition efficiency was also found to be varied with the temperature. The effect of the temperature on corrosion inhibition with and without the inhibitors was studied at different temperatures for the same period of immersion (1 h). As in the table, it is clear that the inhibition efficiency is at its maximum at 28 °C. Above 38 °C, the inhibition efficiency at 200 ppm is almost negligible, but at higher concentrations, it showed a sufficient level of inhibition. Whenever it has a lower efficiency at a normal temperature, it is stable up to higher temperatures also. It is very interesting that the alkaloid was not decomposed even at a temperature of 50 °C. So, it can be claimed that the 800 ppm alkaloids can work at higher temperatures up to 48–50 °C. Because of these results, it can be concluded that the structural deformation of the molecules may occur more often than the desorption of the molecules from the MS surface. This deformation leads to the decrease in the inhibition efficiency

of the inhibitors for the MS in the acidic medium above 58 °C. The inhibition efficiency dependence on the temperature was studied, and it is shown in Figure 7b. The maximum efficiency of 97.27% at the 800 ppm inhibitor at a temperature of 38 °C was achieved.

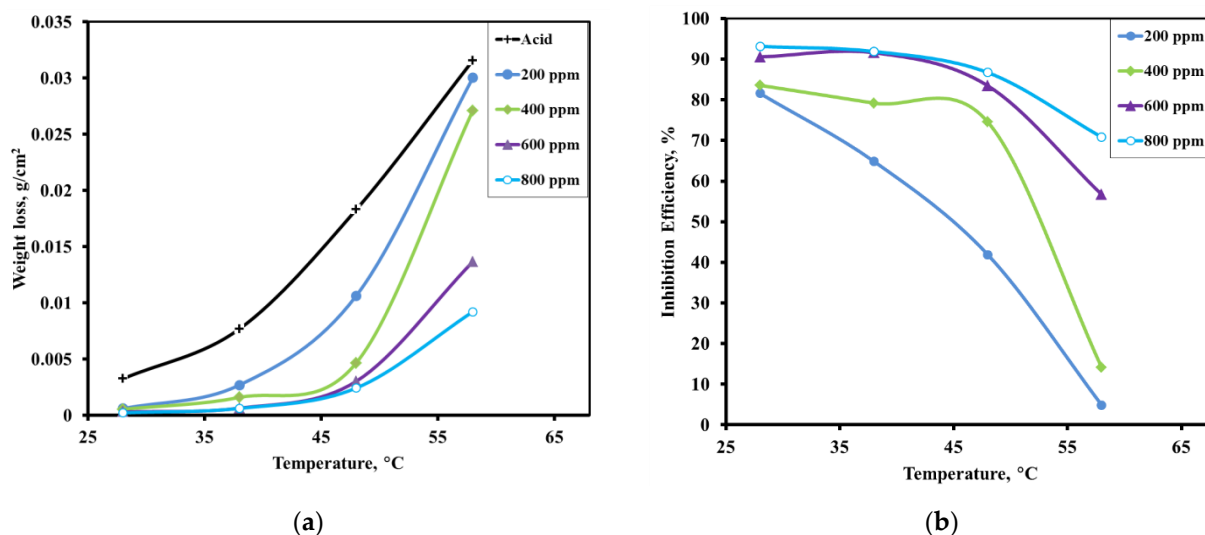


Figure 7. Effect of temperature on (a) weight loss and (b) Inhibition efficiency for the mild steel corrosion in 1 M H₂SO₄ solution immersed for 1 h obtained, which were after taking triplicate measurements ($n = 3$).

3.2.4. Adsorption Isotherm

An adsorption isotherm provides the information regarding the pattern of alkaloid adsorption on the MS surface at the working temperature. Upon immersion, the bulky organic molecules of the inhibitors (alkaloids) become adsorbed onto the MS surface, and they block the active sites of the metal. This restricts both an anodic and a cathodic reaction from occurring between the metal surface and the acid molecule [38,40].

An interesting phenomenon occurs in the adsorption of alkaloids onto the MS surface. When the MS is immersed in the inhibitor solution, firstly, water molecules of the aqueous solution become adsorbed onto the MS surface. Consequently, these adsorbed molecules are replaced by inhibitor molecules, i.e., the quasi-substitution process becomes accelerated [36,37]. The rate of corrosion becomes decreased only when the water molecules on the MS surface are completely replaced by inhibitors. For the study of the adsorption process, three adsorption isotherms: Langmuir, Freundlich, and Tempkin adsorption isotherms, were determined [32,41]. The linear relation between the fraction of the covered surface (θ) and the molar concentration (C_{inh}) had to be established. Khasaine molecules were taken as a reference to calculate the molar concentration of the inhibitor solution [37].

In the study of the Langmuir adsorption isotherm, if the slope of the straight line obtained by plotting $\frac{C_{inh}}{\theta}$ versus C_{inh} as per Equation (4) has unity, it indicates that the monolayer adsorption occurs. The value of the adsorption constant can be obtained from an intercept.

The R^2 values for all of the conditions of C_{inh}/θ versus C_{inh} plot (Figure 8) almost had unity, which indicates the pursuance of the Langmuir model in the adsorption of inhibitor molecules. The free energy of the adsorption was calculated using Equation (5), and it is presented in Table 3.

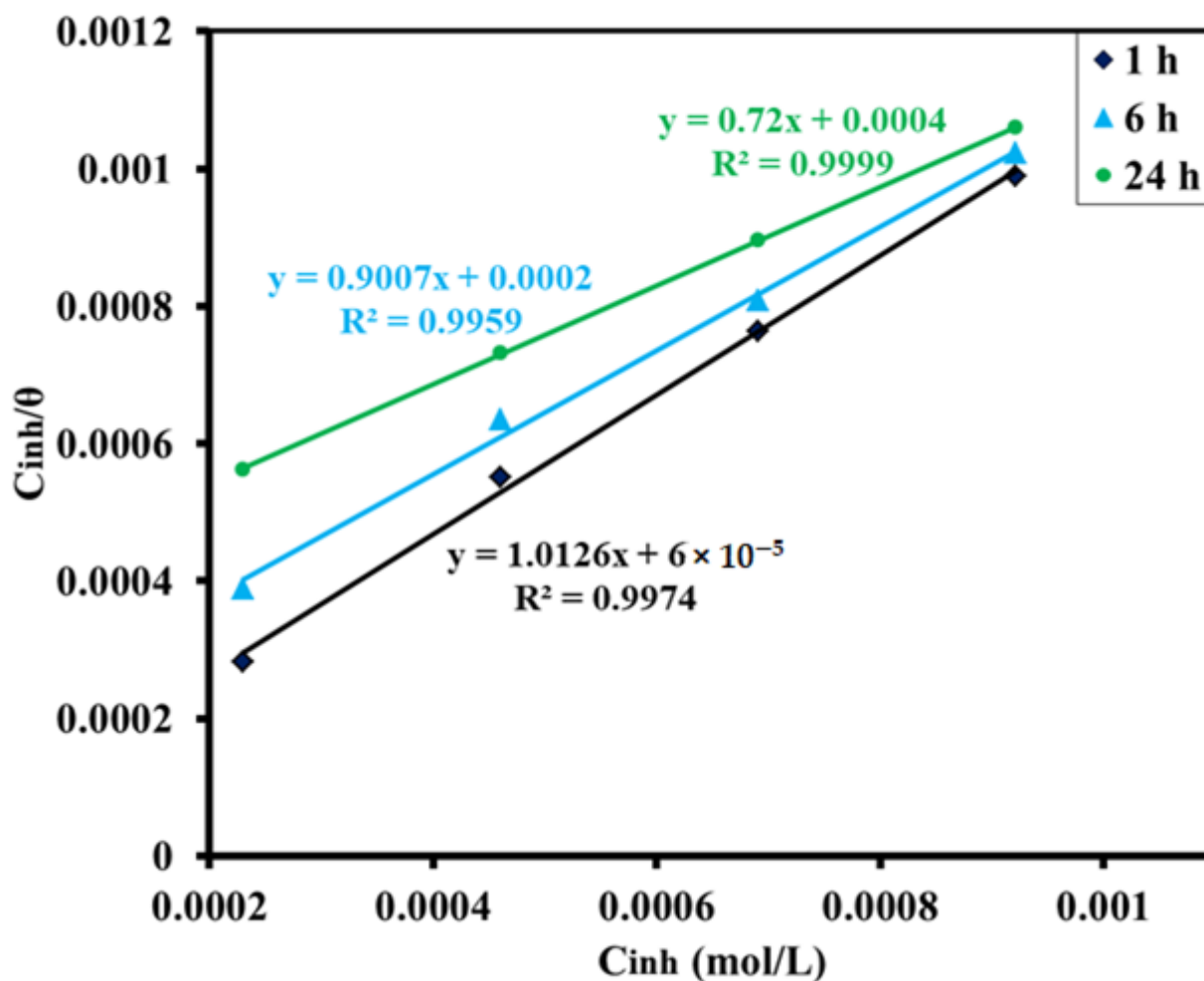


Figure 8. Langmuir adsorption isotherm plot for MS in 1 M H₂SO₄ with different concentration of inhibitor.

Table 3. Different parameters obtained from three different adsorption isotherms.

Isotherm	Graph	Immersion Time (h)	Slope	Intercept	R ²	K _{ads}	−ΔG (kJ/mol)
Langmuir	C/θ vs. C	1	1.0126	0.00006	0.9974	16,666	34.37
		6	0.9007	0.0002	0.9959	5000	31.36
		24	0.72	0.0004	0.9999	2500	29.63
Freundlich	ln θ vs. ln C	1	0.0995	0.616	0.8919		
		6	0.3126	2.0945	0.9909		
		24	0.5477	3.7131	0.9934		
Temkin	θ vs. ln C	1	0.0865	1.5281	0.8872		
		6	0.2296	2.5101	0.9875		
		24	0.3315	3.1834	0.9995		

The inspection of the linear form of the Freundlich adsorption isotherm for the corrosion inhibition study was performed to determine the adsorption easiness. It can be studied to know how easily the adsorption of the inhibitor molecules on the MS surface occurs.

In the study of the Freundlich adsorption isotherm, a graph of ln θ versus ln C was plotted, and the linear correlation coefficient (R²) values of different lines were inspected, as in Figure 9. The details are given in Table 3.

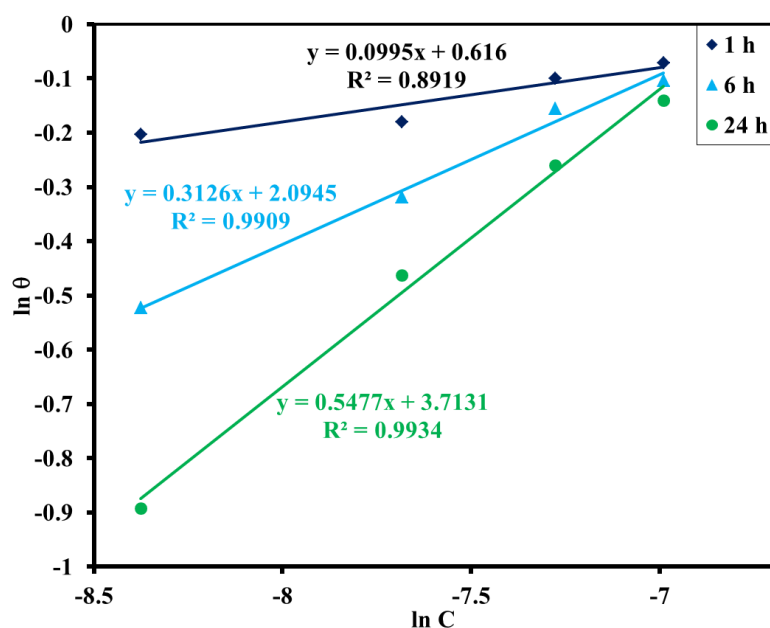


Figure 9. Freundlich adsorption isotherm plot for MS in 1 M H_2SO_4 solution.

The Temkin adsorption isotherm was evaluated in this experiment to evaluate the interaction of the alkaloids with the MS surface. It helps to explain the corrosion inhibition mechanism and the nature of the interaction taking place in the adsorbed layer. A linear form of Equation (7) was used to evaluate the isotherm by plotting θ against $\ln C$.

From the slope of the straight line in Figure 10, the molecular interaction parameter (a) was calculated, and it is tabulated in Table 3. The values of the slope and the intercept have their usual significances. The R^2 value gives the correlation between the measured values, and hence, it helps to assess the linearity range.

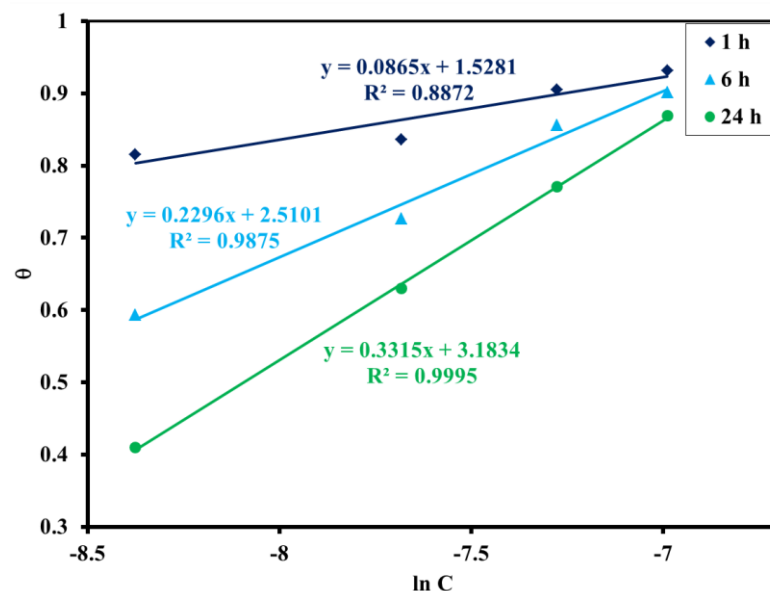


Figure 10. Temkin adsorption isotherm plot for MS in 1 M H_2SO_4 with different concentrations of inhibitor.

The results show that the R^2 values for the Langmuir model nearly have unity at all of the experimented immersion times. This means that the adsorption of the inhibitor onto the MS surface obeys the Langmuir adsorption model for every immersion time. For the

Freundlich and Temkin adsorption isotherms, the correlation coefficient became deviated for the small immersion time, however, it obeys the rule for the prolonged immersion time.

3.2.5. Activation Energy and Corrosion Kinetics

The activation energy of the reaction in the presence and absence of an inhibitor in an electrochemical cell can be explained by rearranging the Arrhenius equation.

Equation (9) reveals that the activation energy of the reaction is equal to the slope of the Arrhenius plot, i.e., a plot obtained between the logarithms of the corrosion rate with $\frac{1}{2.303RT}$ along the axes (Figure 11).

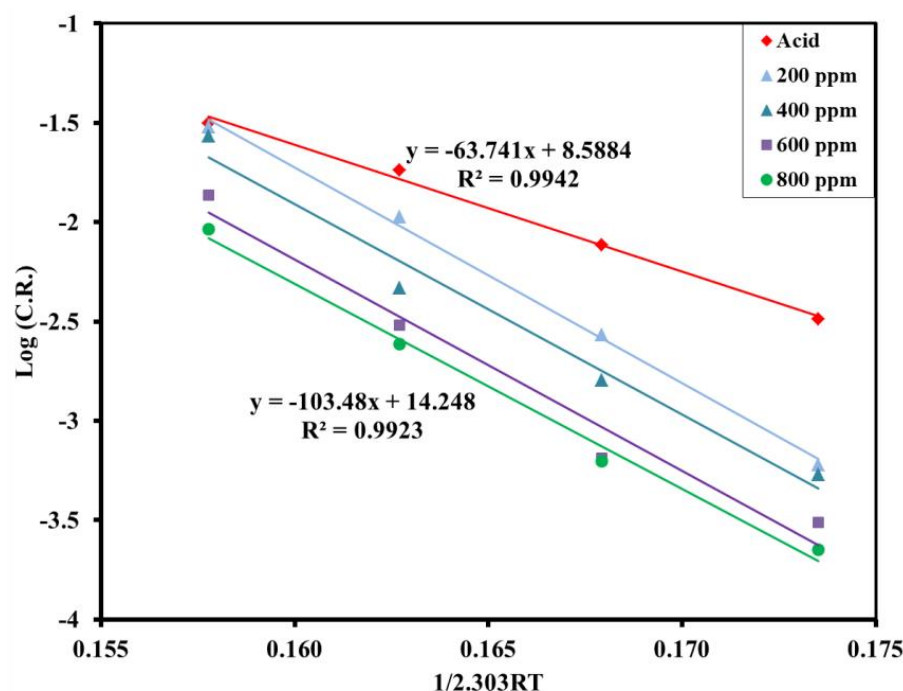


Figure 11. Arrhenius plot for MS in 1M H₂SO₄ with and without inhibitor.

3.2.6. Thermodynamics of Corrosion and Mode of Inhibition

The enthalpy and entropy of the system can be calculated by using the transition state equation, an alternative form of the Arrhenius equation. The enthalpy of activation (ΔH°) is obtained as the slope of a straight line by plotting the $\log \frac{CR}{T}$ versus $\frac{1}{2.303RT}$ in the equation, and the entropy of activation (ΔS°) can be calculated from its intercept (Figure 12). The obtained values of the activation energy are presented in Table 4.

Table 4. Activation parameters of the MS dissolution in 1 M H₂SO₄ without (0 ppm) and with inhibitor.

Inhibitor Solution (ppm)	Log (A)	Ea (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	Ea- ΔH°	ΔS° (J mol ⁻¹ K ⁻¹)
0	8.5884	63.74	61.12	2.62	−89.304
200	15.671	108.72	106.1	2.62	46.298
400	15.044	105.96	103.34	2.62	34.312
600	14.861	106.55	103.92	2.63	30.808
800	14.248	103.48	100.86	2.62	19.071

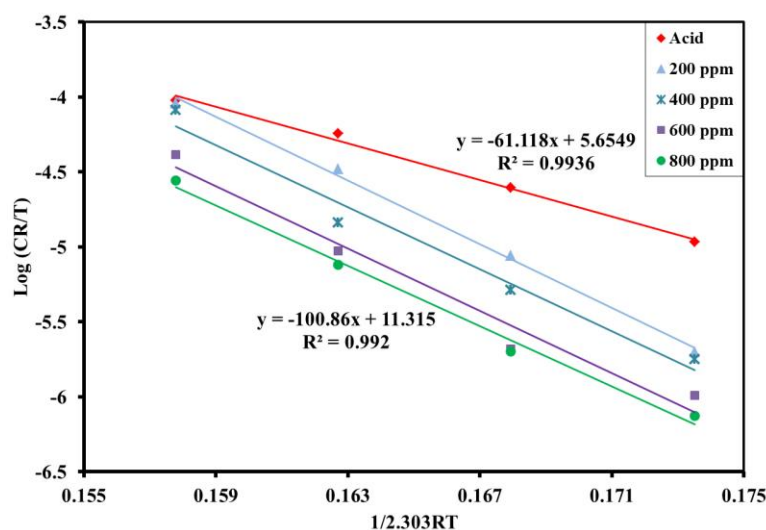


Figure 12. Transition state plot for MS in 1M H₂SO₄ with and without inhibitor.

The nature of the adsorption of the alkaloid molecules on the MS surface could be identified based on the activation energy of the system. An activation energy that is greater than 80 kJ/mol and smaller than 20 kJ/mol of the system indicates chemical and physical adsorption, respectively [42,43]. The energy of activation for the alkaloids are found to be 108.72 kJ/mol in the 200 ppm solution, 105.96 kJ/mol in the 600 ppm solution and 103.46 kJ/mol in the 800 ppm inhibitor solution (Table 4), and they are greater than 80 kJ/mol, indicating strong chemical adsorption. The transition enthalpy of the system is also greater than 100 kJ/mol (positive value), as shown in Table 4. This indicates that the adsorption process is endothermic [44]. Evidently, larger values of transition enthalpy in the presence of the inhibitors than those in the acid confirms that the decrease in the corrosion rate is controlled by kinetic activation [45]. The transition enthalpy value is smaller than that of the activation energy. This insinuates the cathodic hydrogen evolution process (gaseous reaction), which leads to a reduction in the total reaction volume. The activation energy and enthalpy value supports the relation $E_a - \Delta H^\ddagger = RT$ (Table 4) [43,46]. The transition entropy for the acid electrolyte is $-89.30 \text{ Jmol}^{-1}\text{K}^{-1}$ which increases drastically upon the addition of an inhibitor. The transition entropy value is high for all of the concentrations, but it is small for the optimum concentration (800 ppm). This interestingly decreasing value of entropy is also accordance with the decrease in the value of enthalpy of the system. At the optimum concentration, the majority of the MS surface is covered by the inhibitor molecules, and the quasi-substitution process does not occur any longer, which results in the decrease in the entropy. However, the transition entropy of the system is still greater than it is in the acid-only solution, and this could be due to the roaming protons. Very fascinatingly, the increasing transition state entropy of the system is due to the increased density of protons roaming in the solution as well as the quasi-substitution process [7,8].

3.3. Electrochemical Measurement

3.3.1. Open-Circuit Potential Curves

The OCP measurement is one of the simplest concepts for indirect corrosion monitoring. The OCP is the mixed potential that is achieved as a result of the redox reaction. It is also known as the electrode potential when no net current flows through the external circuit of the electrochemical cell. It is not mandatory, but it is generally measured in a three-electrode system by using a potentiostat as a voltage between the working electrode (the metal itself) and the reference electrode (SCE). The variation of the OCP over time can be measured by determining the voltage difference between an MS immersed in a sample solution and as an appropriate reference electrode along with the counter electrode [47].

The variation of the OCP of mild steel immersed in 1 M H_2SO_4 and an inhibitor solution was studied by monitoring the changes in the corrosion potential (Φ_{corr}) over time. The OCP changes of mild steel were measured for 30 min at room temperature for both the immersed and as-immersed conditions.

The OCPs recorded for the as-immersed sample and the immersed sample are shown in Figure 13a,b, respectively. Initially, the potential shifted to a more positive value in both the immersed and as-immersed conditions, indicating that the positive charge is accumulated on the MS surface. The shifting of the potential from the OCP to a more positive value indicates the formation of a protective layer by the inhibitor molecules in the acid solution on the MS surface, (i.e., passivation) which limits the interaction of aggressive ions on the MS surface [13]. The OCP shifting is less than 50 mV, indicating that the alkaloids protect the MS by inhibiting both anodic and cathodic reactions, i.e., mixed inhibition mechanism.

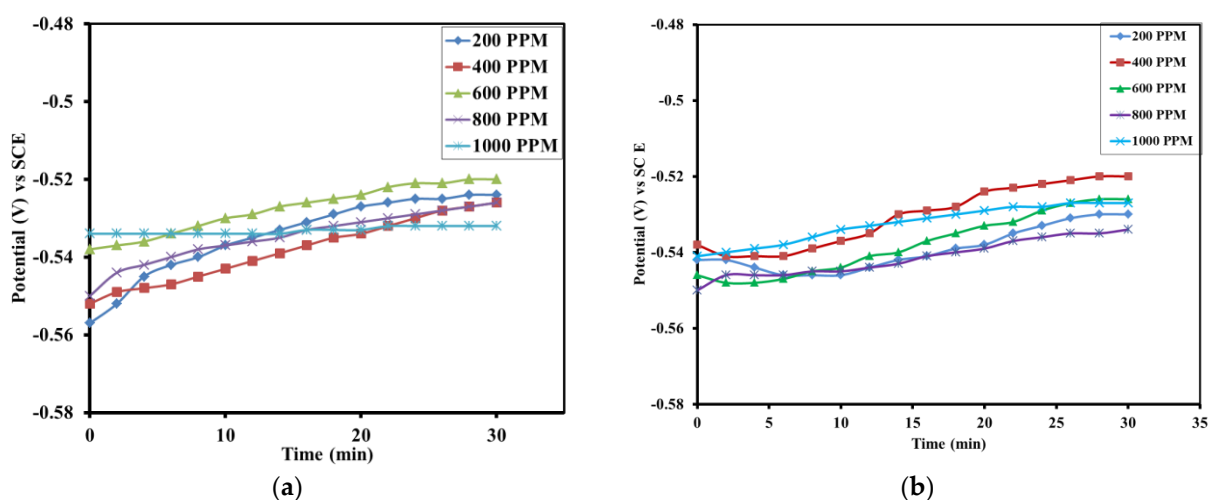


Figure 13. Variation of OCP with the time of immersion of mild steel in different concentrations of inhibitor in 1M H_2SO_4 measured (a) as-immersed condition and (b) after 1 h immersion in solutions.

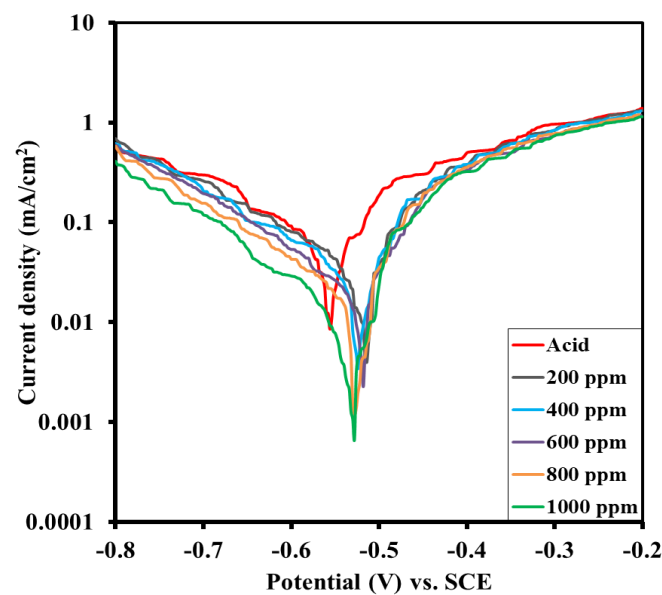
3.3.2. Polarization Curves

The polarization measurement of the MS sample in an as-immersed condition was carried out in a corroding medium in the presence and absence of an inhibitor. In the polarization measurement, different inhibitor concentrations (200, 400, 600, 800 and 1000 ppm) were used and a 300 mV potential was applied for both the anodic and cathodic directions. The corrosion current density in the acid-only solution was found to be 0.0477 mA/cm^2 , which gradually became decreased with the addition of different concentrations of the inhibitors. It decreased up to 0.006 mA/cm^2 for the 1000 ppm inhibitor solution. The current density's decreasing trend is very interesting. It became decreased slightly for the lower concentrations of the inhibitors (200 and 400 ppm), but it was notably for the higher concentrations of the inhibitor (600, 800 and 1000 ppm) solutions. The optimum concentration of the inhibitor for corrosion inhibition in the as-immersed condition was found to be 1000 ppm. All of the data obtained by the polarization measurement for the as-immersed samples are tabulated in Table 5.

The polarization curves (Figure 14) indicate the decrease in the current density after the addition of the inhibitor. It means the MS surface was covered with inhibitor molecules, ensuing fewer chances of having interactions with the corrosive media. This implies that the inhibitor molecules could have created a blockade on the reaction pathway.

Table 5. Table showing OCP, current density, anodic and cathodic slope, and inhibition efficiency for as-immersed sample dipped in acid (0 ppm) and different concentration of inhibitor solutions.

Inhibitor Solution (ppm)	OCP (V)	I _{corr} (mA/cm ²)	Anodic Slope (mA/cm ² /V)	Cathodic Slope (mA/cm ² /V)	Efficiency (%)
0	−0.552	0.0477	12.83	−5.46	
200	−0.521	0.0318	10.87	−5.19	33.33
400	−0.518	0.0209	21.15	−5.7	56.18
600	−0.521	0.016	13.78	−6.52	66.46
800	−0.526	0.0144	17.06	−7.3	69.81
1000	−0.532	0.006	15.01	−4.15	87.42

**Figure 14.** Potentiodynamic polarization curves for mild steel in 1M H₂SO₄ containing different concentrations of inhibitor measured at as-immersed conditions.

The measurement of the corrosion current density and the OCP of the MS samples dipped in acid only and inhibitor solutions were carried out in the potential window from −0.8 to −0.2 V. It was found that the current density for the MS dipped in the acid-only solution is very high, 0.102 mA/cm², but it was decreased drastically for the MS dipped in the inhibitor solutions (Figure 15). The inhibitor efficiency, cathodic and anodic slope, OCP and corrosion current densities obtained by taking potentiodynamic polarization measurements of the MS in the 1 h immersed condition are presented in Table 6.

Table 6. Table showing the OCP, current density, anodic and cathodic slope and inhibition efficiency for immersed sample dipped in acid (0 ppm) and different concentrations of inhibitor solutions.

Inhibitor Solution (ppm)	OCP (V)	I _{corr} (mA/cm ²)	Anodic Slope (mA/cm ² /V)	Cathodic Slope (mA/cm ² /V)	Efficiency (%)
0	−0.545	0.102	15.42	−4.29	
200	−0.52	0.027	10.65	−5.6	73.53
400	−0.529	0.017	14.19	−5.9	83.33
600	−0.526	0.0029	25.26	−6.32	97.16
800	−0.52	0.0025	18.5	−4.9	97.55
1000	−0.537	0.0019	12.5	−4.8	98.14

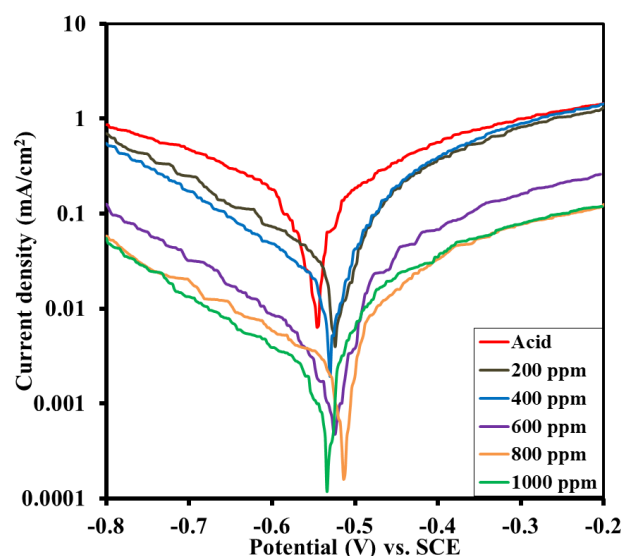


Figure 15. Potentiodynamic polarization curves for mild steel in 1M H₂SO₄ containing different concentrations of inhibitor immersed for 1 h condition.

The decrease in the current density in the presence of an inhibitor is due to the resistance of the inhibitor solution toward corrosion reaction. Hence, like in the as-immersed condition, the current density became decreased due to the coverage of the MS surface by the inhibitor molecules. The nature of the anodic and cathodic slopes were changed for the acid-only and inhibitor solutions, which indicates that the reaction pathways for corrosion inhibition had not been changed.

The inhibition efficiency of the inhibitor in varying concentrations was measured for both the as-immersed and immersed conditions by the polarization method. The inhibition efficiency is very small for a 200 ppm inhibitor solution in the as-immersed condition, but it speeds up and works effectively from the 600 ppm inhibitor solution (Figure 16). It was found that inhibition efficiency increases with an increase in the concentration of the inhibitor solution. This is due to the increase in the fraction of surfaces covered by the inhibitor molecules. The maximum efficiency values were found to be 87.42% and 98.14% at the 1000 ppm concentration of the inhibitor solution in 1M H₂SO₄ for the as-immersed and immersed conditions, respectively.

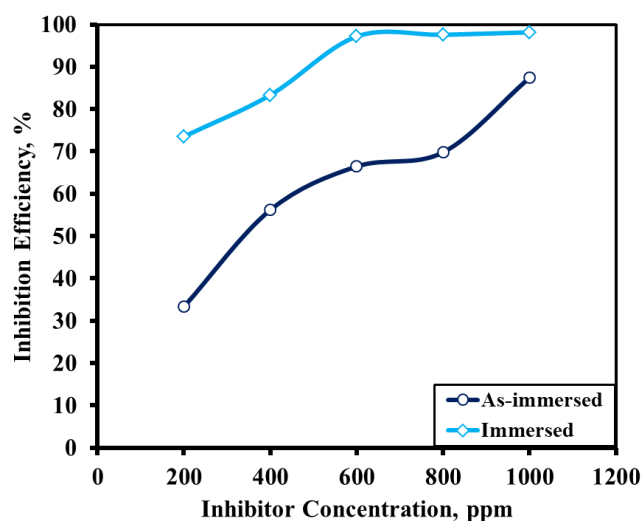


Figure 16. Inhibition efficiency of inhibitor obtained from the polarization of as-immersed and 1 h immersed MS samples in 1M H₂SO₄ in the presence and absence of inhibitor.

3.4. Surface Morphological Study

The optical microscopic images of the MS surfaces dipped in the acid in the absence and presence of the inhibitor solution, as well as a polished sample, are shown in Figure 17. Figure 17a represents the optical image of the polished MS surface, which is clean and very smooth and where no corrosion products appeared. Figure 17b is the image of the MS surface dipped in acid for 1 h, where formation of corrosion products (large pits and rusts) appeared. Figure 17c is the image of the MS surface dipped in the inhibitor solution, where no corrosion products were observed, but small and very interesting greenish colored patches over the MS surface were observed. The absence of corrosion products is due to the presence of an inhibitor that inhibited the acid molecules to react with the MS. Those interesting greenish patches could be the alkaloid molecules adsorbed onto the surface. This indicates that the alkaloids form a protective layer over the MS surface, and hence, they protect it from corrosion [12,13].

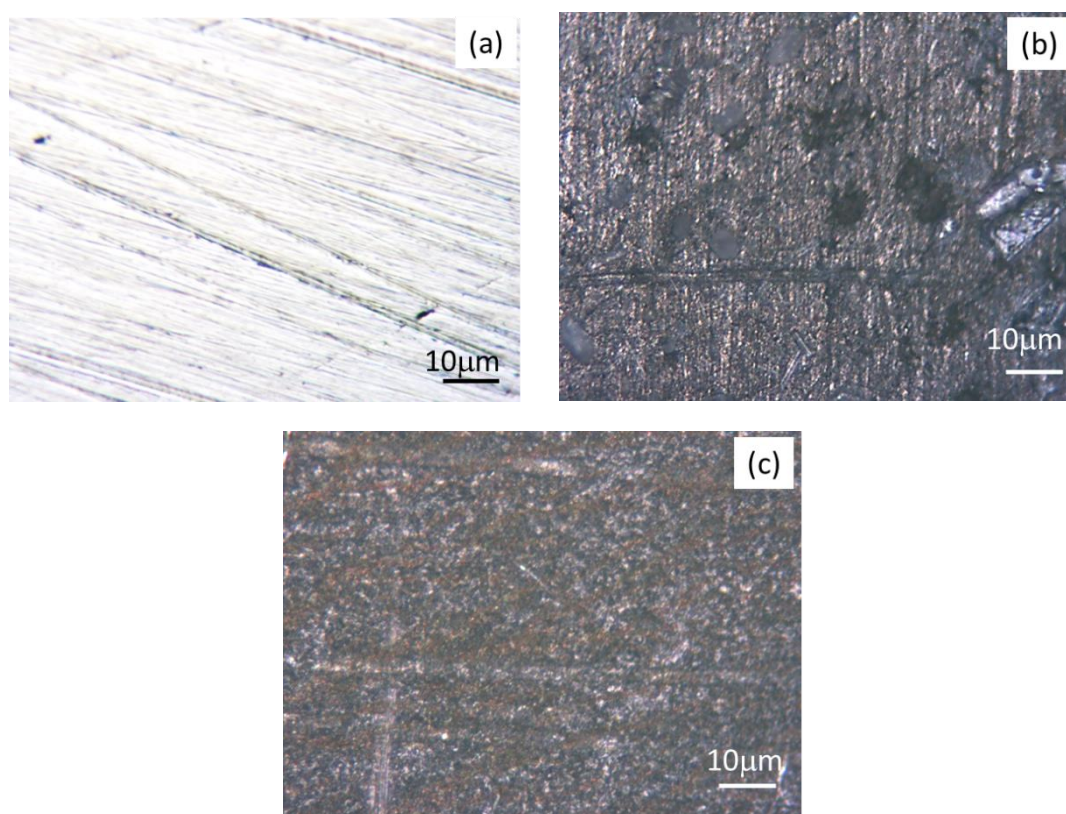
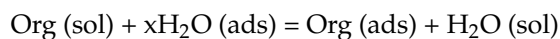


Figure 17. Optical microscopic images of (a) polished MS, (b) MS dipped in acid only and (c) MS dipped in inhibitor solution.

3.5. Mechanism of Corrosion Inhibition

The mechanism behind the corrosion inhibition can be explained based on the above results and discussion. Generally, the green corrosion inhibitor works based on the adsorption mechanism. However, adsorption process in the corrosion inhibition mechanism is not simple. The energy of activation in the acid-only solution is 63.74 kJ/mol, which increases drastically in the presence of alkaloids, and it reached 108.72 kJ/mol in the 200 ppm solution, 105.96 kJ/mol in the 400 ppm solution, and 103.48 kJ/mol in the 1000 ppm inhibitor solution. This reveals that the inhibitors altered the reaction route. However, similar trends in the polarization slope reflect that the reaction pathway has not been changed. So, it is concluded that the inhibitor molecules adsorbed onto the MS surface form a protective layer that increases the activation energy and reduces the corrosion rate by blocking both the anodic and cathodic reactions, simultaneously. The free energy of the adsorption was

found to be intermediate between the physical and chemical adsorption energy, revealing that the physical adsorption was followed by chemical adsorption between the MS and the alkaloids. Corrosion inhibition by alkaloid molecules is due to the adsorption of inhibitor molecules on the MS surface by the replacement of water molecules, which is called the quasi-substitution process.



where Org (sol) and Org (ads) represent the solvated and adsorbed organic (alkaloid) molecules, respectively. Similarly, $\text{H}_2\text{O (ads)}$ represents the adsorbed water molecules on the MS surface, and x represents the size ratio, i.e., many water molecules that are replaced by one organic molecule [17].

The OCP proves the information about where there is a positively or negatively charged MS surface. The OCP of the MS is recorded at around 0.49 V, which is more positive than the PZC value is. It implies that the surface of the metal is positively charged in the inhibitor solution [17,32]. In a positively charged surface, there could be an interaction of SO_4^{2-} ions. Additionally, the surface becomes negatively charged. Alkaloids contain mainly nitrogen as a heteroelement in the ring, which when it is dissolved in an acidic solution, becomes protonated. The protonated alkaloid molecules interact with sulfate ions by the electrostatic force of attraction. The protonated alkaloids return to their neutral form after releasing H_2 molecules [22,32].

Then, the electron pair of HOMO with a high electron density of alkaloids, especially the lone pair of nitrogen, is shared with the vacant d-orbital of iron, forming a coordinate covalent bond, which brings the accumulation of an extra negative charge on the metal surface (Figure 18). To relieve the charge, the electrons are returned to LUMO with high orbital density especially to antibonding π^* orbital of the organic molecule to form a feedback bond. This retrodonation strengthens the bond and inhibitors are adsorbed chemically [13,22,32].

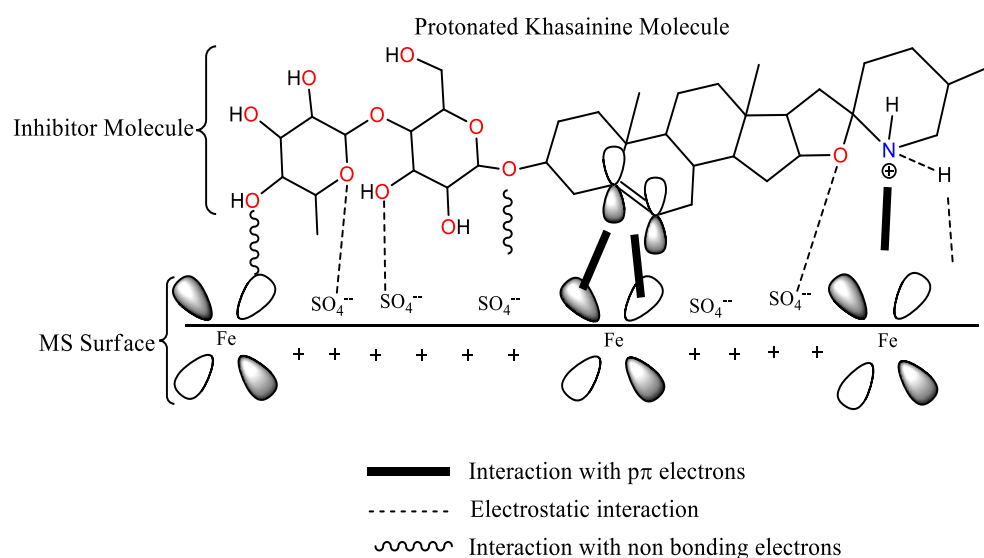


Figure 18. Schematic diagram showing the interaction of active sites of Khasainine molecules with vacant d-orbital of Fe on MS surface.

4. Conclusions

Alkaloids from *Solanum xanthocarpum* stem were successfully extracted, and this was achieved by chemical and spectroscopic test methods. The as-extracted alkaloids were employed as an inhibitor for mild steel corrosion in a 1 M H_2SO_4 solution. The inhibition efficacy of the alkaloids was determined by weight loss and electrochemical (OCP measurement and potentiodynamic polarization) methods at different concentrations (200 ppm, 400 ppm, 600 ppm and 800 ppm) of the inhibitors at different immersion times,

as well as at varied temperatures. The weight loss measurement results reflect that the inhibition efficiency of the alkaloids increased over time, and we found a maximum value of 97.27% for the 800 ppm solution immersed in the medium at 38 °C for 1 h. The polarization experiment revealed that maximum inhibition efficiency was found to be 87.24% for the as-immersed and 98.14% for the 1 h immersed samples in the 1000 ppm alkaloids solutions. From these findings, it can be concluded that *Solanum xanthocarpum* alkaloids can be used as an excellent, eco-friendly green inhibitor for mild steel corrosion.

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